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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Kambe et al.

Serial No.: 08/962,362

Filed : October

: October 31, 1997

For

PHOSPHORS

Docket No.: N19.12-0006

Group Art Unit:

2879

Examiner: M. Day

DECLARATION UNDER 37 C.F.R. § 1.132

Express Mail: EL418983858US
Date of Deposit: March 24, 2000

Assistant Commissioner for Patents Washington, D.C. 20231

I, Rajiv K. Singh, Ph.D., hereby declare as follows:

- I am presently a Professor of Material Science and Engineering at the University of Florida at Gainsville. Also, I am also Director of the Characterization, Research Instrumentation and Testbed Facility of the Engineering Research Center for Particle Science and Technology at the University of Florida. Apart from my academic responsibilities, I provide consulting services through R. K. Singh Consulting Inc.
- 2. I received my Ph.D. degree in 1989 in Material Science and Engineering from North Carolina State University, Raleigh, NC.
- I have been on the faculty at the University of Florida since 1990. I was promoted to Associate Professor with tenure in 1995 and to full Professor in 1997. A copy of my Curriculum Vitae is attached.
- 4. My recent accomplishments include receiving a National Science Foundation Young Investigator Award in 1994 and the Hardy Gold Metal for Outstanding Contributions in Material Science in 1995. I was a Distinguished Visiting Professor/Scientist at National University of Singapore (1999) and National Institute for Materials and Chemical Research, Tsukuba, Japan (2000). I am the

author or co-author of more than 293 scientific articles and conference proceedings. I have co-edited five books and guest edited five journal issues.

- 5. I am under a Consulting Agreement with NanoGram Corporation to provide consulting services in the area of chemical-mechanical planarization. I am not a shareholder in NanoGram Corporation.
- I have read carefully the pending claims of the above noted patent application entitled "PHOSPHORS" and U.S. Patent 5,442,254 to Jaskie (the Jaskie patent). I did not participate in any capacity with the preparation of the PHOSPHORS patent application.
- 7. I am very familiar with approaches that have been attempted for separating nanoparticles by filtration. knowledge, no experimental results based on the separation of nanoparticles, by the wet filtration approaches described in the Jaskie patent at column 7, lines 28-40 have ever been reported in the public literature. Since such chromatography techniques are not known for the separation of nanoparticles, a person of skill in the art would expect to expend a substantial amount of inventive effort to attempt to practice the wet filtration techniques. Without at least some preliminary results to support the basic principles underlying the effort, there would be no reasonable expectation of eventual success at applying the wet filtration approach. In my opinion, the wet filtration approaches described the Jaskie patent at column 7, lines 28-40 are highly speculative, and the description in the Jaskie patent does not provide a reasonable expectation of successfully separating a collection of nanoparticles to isolate a particle population with a desired narrow range of particle sizes.
- 8. Since the Jaskie wet filtration techniques would be difficult or impossible to scale up to commercial quantities, it is unlikely that any effort will ever be spent on developing such approaches. The only possibility for the expenditure of experimental effort on such wet filtration techniques would be to

satisfy academic curiosity. In the biological sciences, gel electrophoresis is an important technique for the characterization of biochemical preparations. However, there are other long established approaches for characterizing nanoparticles.

- I am aware of considerable amounts of effort expended using conventional size exclusion filtration for the preparation of nanoparticle collections. At the time of filing the PHOSPHORS patent application on October 31, 1997, there were no filtration approaches publicly known that could create a collection of phosphorescent nanoparticles with a very narrow size distribution as disclosed and claimed in the PHOSPHORS patent application. At best, these filtration techniques could only exclude micron scale
- 10. Based on my extensive knowledge in the nanoparticle it is my opinion that the "tuning (size selection)" described in the Jaskie patent at column 7, lines 28-30 could not be accomplished based on publicly available filtration methods as of October 31, 1997. Specifically, tuning could not be performed by the wet filtration approach outlined in the Jaskie patent without the expenditure of an undue amount of experimentation, if
- 11. I declare that all statements made herein that are of my own knowledge are true and that all statements that are made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: March 21'00 By: Ray K. Ly

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the presence of the electric field. While gel electrophoresis is used to separate biological macromolecules using electric fields, these separation are performed in polymer gels, not cloth, that have been developed for the specific purpose of separating biological macromolecules. The gel acts as a sieve. allowing the fractionation of the biological macromolecules by size, surface charge and steric properties. No cloth exists with the required sieving properties. Even the protocols effective to separate different types of biological macromolecules, such as water soluble proteins, membrane proteins and nucleic acid fragments, are significantly different from each other. Inorganic particles are very different with respect to chemical properties chemical structure from biological macromolecules. The Jaskie patent provides no information that guides anyone trying to adapt these biochemical methods to the separation of quantum particles. Thus, a person familiar with the separation technologies could not separate Jaskie's quantum particles based on information provided in the Jaskie patent.

I declare that all statements made herein that are of my own knowledge are true and that all statements that are made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: 10/8/00

Terry M. Bricker, Ph.D.

IN THE UNITED STATES PATERTIAND TRADEMARK OFFICECHNOLOGY CONTER

Applicant : Kambe et al.

Serial No.: 08/962,362

Filed

:October 31, 1997

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: PHOSPHORS

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Group Art Unit: 2879

Examiner: M. Day

DECLARATION UNDER 37 C.F.R. § 1.132

Express Mail: EL 636050305UJ Date of Deposit: October 10, 2000

BOX AF Assistant Commissioner for Patents Washington, D.C. 20231

I, Terry M. Bricker, Ph.D., hereby declare as follows:

- I am presently the Mooreland Family Professor of Basic Sciences, Department of Biological Sciences and an Professor of Chemistry at Louisiana State University, Baton Rouge, Louisiana.
- 2. I received my Ph.D. degree in 1981 in Botany from Miami University.
- 3. I have been on the faculty at the Louisiana University I was promoted to Associate Professor with tenure in 1990 and to full Professor in 1994. A copy of my Resume is attached.
- I have been Visiting Professor at the University of Illinois and Michigan State University. I am the author or coauthor of many scientific articles, conference proceedings and review papers. I have served on the editorial board of Plant Physiology and the Annual Reviews of Plant Physiology and Plant Molecular Biology.
- 5. I have no financial interest in NanoGram Corporation or in the present patent application.

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- I have extensive experience in separation technology applied to biological nanoparticles, in particular, proteins and DNA. I and coworkers in my laboratory continuously use several forms of chromatography in the separation and purification of proteins. I have used chromatography and protein purification techniques throughout my career.
- 7. I have read carefully U.S. Patent 5,442,254 to Jaskie (the Jaskie patent). I have evaluated the description of particle separation in the Jaskie patent based on my extensive expertise in separation technology generally.
- 8. The isolation method of the quantum particles describe in the Jaskie patent at column 7, lines 28-40 relies on the use of capillary action to separate particles of different diameters. Specifically, particles with a diameter range of about 10 to 100 angstroms are suspended in a liquid, and the liquid is allowed to move up a cloth by capillary action. The authors argue that the distance which various particles migrate up the cloth is directly proportional to their size. The authors further suggest that at any given height up the cloth all of the particles will be the same size. Thus, the authors are describing a chromatographic system which they allege will differentially fractionate the particles based on size.
- 9. separation techniques described in the Jaskie patent will not separate different size classes of quantum First, a mixture of different sized particles is particles. continuously loaded onto the cloth. Even assuming for argument that the different sized particles migrate at different speeds, additional particles are continuously loaded behind the migrating edge of initially loaded particles. Thus, the particles are continuously remixed with particles of other sizes as additional particles are loaded onto the cloth. This remixing occurs for every size class of particles. Significantly, the technique will not work because no cloth is known with the necessary properties to differentially interact with different sized particles.

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Traditional thin layer chromatography is based on differential solubility constants for the chemicals being separated. Due to different solubility constants, solutes migrate at different rates as the solvent is taken up by capillary action.

- 10. Any chromatographic separation approach relies on 1) the properties of the liquid that the particles are suspended in, 2) the surface characteristics of the cloth, 3) the surface properties of the quantum particles, and 4) the size of the quantum particles. The patentees provide no direct information on the first three of these categories. Based on the discussion in the previous paragraph in column 7 of the patent, perhaps one can assume that the solvent is water. However, the surface properties of the cloth used in the described separation is critical. Separation in chromatographic any system is dependent differential partitioning of the solutes, i.e., the particles, between a mobile phase, the water, and a stationary phase, the cloth. However, no such cloth exists. For separation of biological macromolecules, such separation by size is the purview of gel filtration chromatography. Even in gel filtration fractionation systems, the relatively small differential partitioning coefficients observed prevent true high resolution separations. In this particular instance, one must necessarily obtain high degrees of dimensional resolution of the quantum particles to achieve wavelength selectivity. The dimensional resolution cannot be achieved by the methods presented in column 7, lines 28-40 of the Jaskie patent.
- 11. The addition of an electric field would not overcome these problems. First, the authors do not make any claim that the surface charge on the particles is directly proportional to the size. The authors do not describe the means of attaching the electric current or even if the current is applied axially of perpendicular to the capillary flow. Critically, the authors do not describe the properties of the cloth even though the properties of the cloth would critically effect the separation in